

Supporting Information

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**Asymmetric Tandem Wittig Rearrangement/Mannich Reactions\*\***

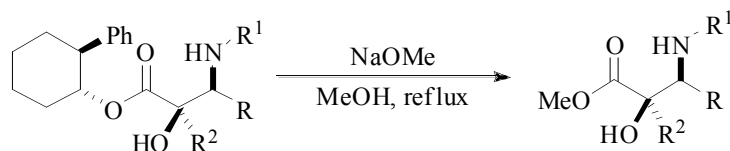
*Natalie C. Giampietro and John P. Wolfe\**

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**General:** All reactions were carried out under a nitrogen atmosphere in oven- or flame-dried glassware. Dibutylboron triflate (1.0 M solution in dichloromethane) was purchased from Aldrich Chemical Co. and was used as obtained. All imines and  $\alpha$ -amido sulfones were prepared from aldehydes obtained from commercial sources (Aldrich Chemical Co. or Acros Chemical Co.). Aldehydes were purified by distillation from crushed anhydrous  $\text{Ca}_2\text{SO}_4$ . Triethylamine and diisopropylethylamine were obtained from Aldrich Chemical Co. and were purified by distillation from  $\text{CaH}_2$ . Phosphate buffer solution (pH 7) and 2-(benzyloxy)acetyl chloride were obtained from commercial sources and were used as obtained. *N*-(benzylidene)benzylamine,<sup>1</sup> *N*-(4-methoxybenzylidene)benzylamine,<sup>1</sup> *N*-(furylidene)benzylamine,<sup>1</sup> *N*-(4-fluorophenylmethylidene)benzylamine,<sup>1</sup> *N*-*tert*-butoxycarbonyl- $\alpha$ -(phenylsulfonyl)benzylamine,<sup>2</sup> *N*-(*tert*-butoxycarbonyl)benzylamine,<sup>2</sup> *N*-*tert*-butoxycarbonyl-3-methyl-1-(phenylsulfonyl)butylcarbamate,<sup>3</sup> *N*-*tert*-butoxycarbonyl- $\alpha$ -cyclohexyl(phenylsulfonyl)methylcarbamate,<sup>3</sup> *N*-(benzylidene)-4-methoxyaniline,<sup>4</sup> *N*-benzylideneaniline,<sup>4</sup> *N*-(cyclohexylmethylidene)benzylamine,<sup>5</sup> (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate,<sup>6</sup> and (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(allyloxy)acetate<sup>6</sup> were prepared according to published procedures. Methylene chloride, tetrahydrofuran, and ether were purified using a GlassContour solvent purification system. Yields refer to isolated yields of compounds estimated to be  $\geq 95\%$  pure as determined by  $^1\text{H}$  NMR, GC, and/or combustion analysis. The yields reported in the Supporting Information describe the result of a single experiment, whereas the yields reported in Tables 1–3 and eq 2–6 are average yields of two or more experiments. Thus, the yields reported in the Supporting Information may differ from those shown in Tables 1–3 and eq 2–6.

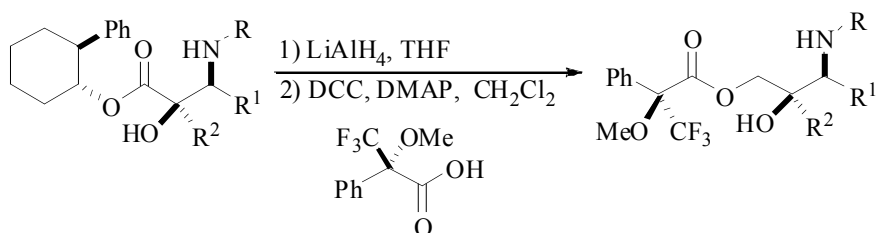
**General Procedure A: Tandem Wittig rearrangement/Mannich reactions.** A flame-dried flask was cooled under a stream of nitrogen and charged with a 1 M solution of dibutylboron triflate in dichloromethane (4.0 equiv). The pale yellow solution was cooled to 0 °C, and triethylamine or diisopropylethylamine (3.2 equiv) was added dropwise to afford a colorless solution. A solution of the ester substrate (1 equiv) in  $\text{CH}_2\text{Cl}_2$  (1 mL/mmol substrate) was then added dropwise, and the reaction mixture was warmed to rt, stirred for 15 min (with triethylamine as base) or 20 min (with diisopropylethylamine as base), and then cooled to 0 °C. A solution of the imine (1.5 or 2 equiv) or  $\alpha$ -amido sulfone (2 equiv) in  $\text{CH}_2\text{Cl}_2$  (1 mL/mmol

substrate) was added dropwise, and the reaction mixture was warmed to rt and stirred for 3–12 h. The reaction vessel was then opened to air, and pH 7 buffer (1 mL/0.1 mmol substrate), and methanol (2 mL/0.1 mmol substrate) were added. The resulting mixture was cooled to 0 °C, 30% aqueous H<sub>2</sub>O<sub>2</sub> (2 mL/0.1 mmol substrate) was added slowly, and the reaction mixture was warmed to rt and stirred for 1 h. The mixture was diluted with ether (10 mL/0.1 mmol substrate) and water (5 mL/0.1 mmol substrate), then was transferred to a separatory funnel. The layers were separated, and the organic layer was washed with a saturated aqueous solution of FeSO<sub>4</sub> (4 x 5 mL/0.1 mmol substrate) until a red-orange aqueous phase no longer persisted in order to quench any remaining peroxide. *Caution! This procedure is exothermic. The FeSO<sub>4</sub> solution should be added via glass pipette SLOWLY DROPWISE.* The organic layer was then washed with brine, dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography on silica gel.



**General Procedure B: Method to Assess Enantiopurity of Products via Conversion to Methyl Esters (19, S1, S3).** In order to assess the enantiomeric purity of the products formed in the tandem Wittig-rearrangement/Mannich reactions, the 2-phenylcyclohexyl esters **4–5**, and **7** were converted to the corresponding methyl esters **19**, **S1** and **S3** using the following procedure. The glycolate ester (1.0 equiv) was dissolved in methanol (0.1 M) and added to a flame-dried 2-neck flask fitted with a reflux condenser under nitrogen. A solution of NaOMe (4.0 M in MeOH) was added, and the reaction mixture was heated to reflux and stirred until the starting material had been completely consumed as judged by TLC analysis (ca. 2 h). The reaction mixture was cooled to rt, quenched with 1 M HCl (1 mL/0.1 mmol substrate) and concentrated *in vacuo*. The crude residue was diluted with H<sub>2</sub>O (1 mL/0.1 mmol substrate), and extracted with Et<sub>2</sub>O (3 x 2 mL/0.1 mmol substrate). The phases were separated, and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude methyl ester product was purified by flash chromatography on silica gel. The enantiopurity was subsequently determined by chiral HPLC analysis.

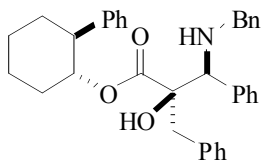
For purposes of comparison, racemic amino-alcohol methyl esters were prepared from the corresponding *O*-benzyl or *O*-allyl methyl esters,<sup>7</sup> using a procedure identical to that described above.



**General Procedure C: Method to Assess Enantiopurity of Products (S4, S4-S12) via Reduction to Triol and Conversion to Mosher Ester.** In order to assess the enantiomeric purity of the products formed in the tandem Wittig-rearrangement/Mannich reactions, the 2-phenylcyclohexyl esters **6**, **8–11**, **13–14**, and **16–18** were converted to the corresponding Mosher esters **S2**, and **S4–S12** using the following procedure. The glycolate ester (1.0 equiv) was dissolved in THF (0.1 M) and cooled to 0 °C. A solution of LiAlH<sub>4</sub> (2 equiv/mmol substrate, 1.0 M in THF) was added, the reaction mixture was warmed to rt, and stirred until the starting material had been completely consumed as judged by TLC analysis. The reaction mixture was cooled to 0 °C and quenched with H<sub>2</sub>O (1 mL/0.1 mmol substrate). The crude residue was diluted with Et<sub>2</sub>O (2 mL/0.1 mmol substrate), 10 M NaOH was added (1 mL/0.1 mmol substrate), then H<sub>2</sub>O (0.5 mL/0.1 mmol substrate) was added. The phases were separated, the inorganic precipitate was washed with ether (3 x 2 mL), and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude triol product was purified by flash chromatography on silica gel.

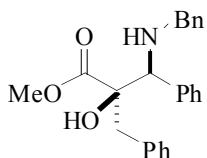
A solution of the triol in CH<sub>2</sub>Cl<sub>2</sub> (0.2 M), DCC (1.1 equiv), DMAP (0.2 equiv) and (–)-(S)-3,3,3-trifluoro-2-methoxy-2-phenylpropanoic acid (1.1 equiv) were combined and stirred at room temperature until the triol had been completely consumed as judged by TLC analysis. The reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub>, filtered through a cotton plug, and concentrated *in vacuo*. The crude product was purified by flash chromatography on silica gel. The enantiopurity was subsequently determined by <sup>19</sup>F NMR analysis.

For purposes of comparison, racemic amino-alcohol Mosher esters were prepared from the corresponding *O*-benzyl or *O*-allyl methyl esters,<sup>7</sup> using a procedure identical to that described above.

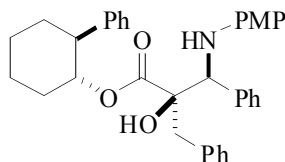


**(-)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-benzyl-3'-benzylamino-2'-hydroxy-3'-phenylpropanoate (4).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (47 mg, 0.14 mmol) with *N*-(benzylidene)benzylamine<sup>1</sup> (42 mg, 0.22 mmol) was conducted according to General Procedure A using triethylamine as base to afford 54 mg (72%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_D^{23}$  -36.8 (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.23–7.24 (m, 8 H), 7.21–7.14 (m, 6 H), 7.13–7.11 (m, 3 H), 7.08–7.05 (m, 3 H), 4.81–4.76 (m, 1 H), 3.48 (s, 1 H), 3.30 (s, 1 H), 3.03 (d, *J* = 13.5 Hz, 1 H), 2.86–2.80 (m, 2 H), 2.60 (d, *J* = 13.0 Hz, 1 H), 2.53 (d, *J* = 13.5 Hz, 1 H), 2.06–2.03 (m, 1 H), 1.95–1.92 (m, 1 H), 1.80–1.77 (m, 2 H), 1.64–1.54 (m, 2 H), 1.44–1.36 (m, 2 H), 1.26–1.23 (m, 1 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  174.6, 143.5, 140.8, 138.9, 136.1, 130.4, 128.7, 128.6, 128.1, 128.0, 127.9, 127.7, 127.5, 126.7, 126.5, 80.3, 80.1, 68.1, 50.4, 49.9, 42.4, 34.0, 31.9, 25.8, 24.6 (two carbon signals are absent due to incidental equivalence); IR (film) 3494, 2931, 1725 cm<sup>-1</sup>. MS (ESI) 520.2835 (520.2852 calcd for C<sub>35</sub>H<sub>37</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding methyl ester (**19**) through reaction with NaOMe using General Procedure B. This procedure afforded 27 mg (62%) of **19**. The enantiopurity of the methyl ester was determined to be 96% ee by chiral HPLC analysis (chiracel AD 0.46 cm x 15 cm, 10% isopropanol/ hexanes, 0.5 mL/min, RT= 23.9 and 28.4 min).

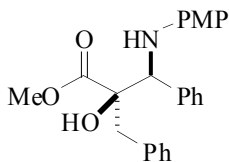


**(+)-(2*R*,3*S*)-Methyl-2-benzyl-3-benzylamino-2-hydroxy-3-phenylpropanoate (19).**  $[\alpha]_D^{23} +30.9$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.45–7.37 (m, 3 H), 7.37–7.27 (m, 4 H), 7.24–7.16 (m, 6 H), 7.01–7.00 (m, 2 H), 3.90 (s, 1 H), 3.72 (s, br, 1 H), 3.69 (s, 3 H), 3.46 (s, 1 H), 3.37 (d, *J* = 13.5 Hz, 1 H), 2.78 (d, *J* = 13.0 Hz, 1 H), 2.40–2.37 (m, 2 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  175.6, 140.2, 138.4, 135.6, 128.8, 129.3, 128.4, 128.3, 128.1, 128.0, 127.8, 126.9, 126.7, 81.7, 65.9, 52.5, 50.1, 43.2; IR (film) 3502, 3029, 1738 cm<sup>-1</sup>. MS (ESI) 376.1898 (376.1913 calcd for C<sub>24</sub>H<sub>25</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).



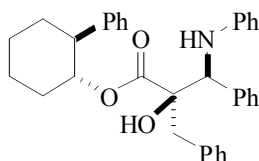
**(-)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-benzyl-2'-hydroxy-3'-(*p*-methoxyphenylamino)-3'-phenylpropanoate (5).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-benzyloxyacetate (**3a**) (67 mg, 0.21 mmol) with *N*-(benzylidene)-4-methoxyaniline<sup>4</sup> (66 mg, 0.31 mmol) was conducted according to General Procedure A using triethylamine as base to afford 97 mg (87%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 4:1 dr following purification.  $[\alpha]_D^{23} -35.9$  (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.18–7.08 (m, 12 H), 7.04–7.02 (m, 3 H), 6.56–6.54 (m, 2 H), 6.13–6.11 (m, 2 H), 4.96–4.90 (m, 1 H), 3.40 (s, br, 1 H), 4.13–4.11 (m, 1 H), 3.68 (s, 3 H), 3.31 (s, 1 H), 2.85–2.78 (m, 2 H), 2.68–2.65 (m, 1 H), 2.00–1.90 (m, 2 H), 1.80–1.73 (m, 2 H), 1.54–1.44 (m, 2 H), 1.40–1.23 (m, 2 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  174.1, 151.8, 142.6, 140.1, 138.2, 135.4, 130.4, 128.6, 128.5, 128.0, 127.9, 127.4, 127.0, 126.8, 126.8, 115.6, 114.3, 79.6, 79.5, 62.9, 55.6, 49.5, 42.7, 34.8, 31.9, 25.7, 24.6; IR (film) 3378, 2931, 1733 cm<sup>-1</sup>. MS (ESI) 558.2617 (558.2620 calcd for C<sub>35</sub>H<sub>37</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding methyl ester (**S1**) through reaction with NaOMe using General Procedure B. The enantiopurity of the methyl ester was determined to be 58% ee by chiral HPLC analysis (chiracel AD 0.46 cm x 15 cm, 10% isopropanol/ hexanes, 1.0 mL/min, RT= 19.3 and 31.9 min).



**(-)-(2R,3S)-Methyl-2-benzyl-2-hydroxy-3-(*p*-methoxyphenylamino)-3-phenylpropanoate**

**(S1).**  $[\alpha]_D^{23}$  -4.6 (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.45–7.43 (m, 2 H), 7.36–7.31 (m, 2 H), 7.28–7.17 (m, 4 H), 7.07–7.04 (m, 2 H), 6.68–6.63 (m, 2 H), 6.55–6.52 (m, 2 H), 4.78 (s, 1 H), 4.59 (s, 1 H), 3.71 (s, 3 H), 3.67 (s, 3 H), 3.41 (s, 1 H), 3.01 (d, *J* = 13.2 Hz, 1 H), 2.52 (d, *J* = 13.2 Hz, 1 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  175.0, 140.3, 138.1, 135.3, 129.8, 128.9, 128.2, 128.1, 127.8, 126.9, 115.7, 114.6, 81.1, 63.6, 55.6, 53.0, 43.4, 27.0; IR (film) 3503, 2931, 1735 cm<sup>-1</sup>. MS (ESI) 414.1673 (414.1681 calcd for C<sub>24</sub>H<sub>25</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

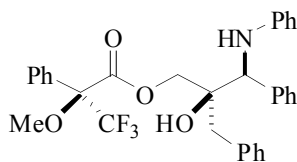


**(-)-(1R,2S,2'R,3'S)-2-Phenylcyclohexyl-2'-benzyl-2'-hydroxy-3'-phenylamino-3'-phenylpropanoate (6).**

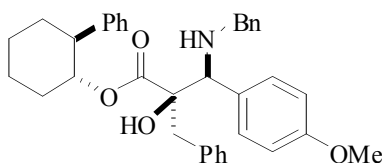
The reaction of (-)-(1R,2S)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (45 mg, 0.14 mmol) with *N*-benzylideneaniline<sup>4</sup> (37 mg, 0.21 mmol) was conducted according to General Procedure A using triethylamine as base to afford 47 mg (67%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 11:1 dr following purification.  $[\alpha]_D^{23}$  -63.8 (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.20–7.16 (m, 8 H), 7.15–7.12 (m, 2 H), 7.10–7.07 (m, 2 H), 7.05–6.99 (m, 3 H), 6.98–6.93 (m, 2 H), 6.59–6.53 (m, 1 H), 6.21–6.20 (m, 2 H), 4.96–4.91 (m, 1 H), 4.67 (d, *J* = 10.5 Hz, 1 H), 4.20 (d, *J* = 10.0 Hz, 1 H), 3.29 (s, 1 H), 2.86 (s, 1 H), 2.84–2.79 (m, 1 H), 2.70–2.67 (m, 1 H), 1.99–1.91 (m, 2 H), 1.79–1.74 (m, 2 H), 1.53–1.43 (m, 1 H), 1.40–1.33 (m, 2 H), 1.30–1.22 (m, 1 H); <sup>13</sup>C NMR

(125 MHz, CDCl<sub>3</sub>)  $\delta$  174.0, 145.9, 142.5, 138.0, 135.2, 130.4, 128.7, 128.6, 128.0, 127.9, 127.5, 127.0, 126.9, 126.8, 117.2, 114.3, 79.6, 79.5, 62.9, 49.5, 42.6, 34.7, 31.9, 25.7, 24.6 (one carbon signal is absent due to incidental equivalence); IR (film) 3396, 2924, 1717 cm<sup>-1</sup>. MS (ESI) 528.2524 (528.2515 calcd for C<sub>34</sub>H<sub>35</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S2**) using General Procedure C. This procedure afforded 17 mg (57%) of **S2**. The enantiopurity was determined to be 83% ee by <sup>19</sup>F NMR analysis.



**(-)-(1*S*,2'*R*,3'*S*)-2'-Benzyl-3'-benzylamino-3'-furan-2-yl-2-hydroxy-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S2**).** [ $\alpha$ ]<sub>D</sub><sup>23</sup> -30.6 (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.54–7.52 (m, 2 H), 7.40–7.36 (m, 1 H), 7.34–7.29 (m, 3 H), 7.28–7.23 (m, 7 H), 7.07–6.97 (m, 2 H), 6.99–6.97 (m, 1 H), 6.67–6.62 (m, 2 H), 6.50–6.48 (m, 2 H), 4.89–4.86 (m, 1 H), 4.57–4.55 (m, 1 H), 4.41–4.38 (m, 1 H), 3.92–3.90 (m, 1 H), 3.57 (s, 3 H), 2.77 (d, *J* = 14.0 Hz, 1 H), 2.56–2.53 (m, 1 H), 1.54 (s, 1 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO)  $\delta$  -70.9; IR (film) 3402, 2924, 1750 cm<sup>-1</sup>. MS (ESI) 572.2034 (572.2025 calcd for C<sub>31</sub>H<sub>30</sub>F<sub>3</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

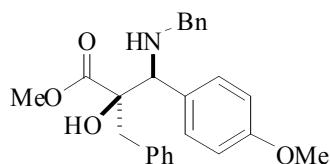


**(-)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-benzyl-3'-benzylamino-2'-hydroxy-3'-*p*-methoxyphenylpropanoate (**7**).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (56 mg, 0.17 mmol) with *N*-(4-methoxybenzylidene)benzylamine<sup>1</sup> (58 mg, 0.26 mmol) was conducted according to General Procedure A using triethylamine as base to afford 87 mg (93%) of the title compound as a colorless oil. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 20:1 dr following purification. [ $\alpha$ ]<sub>D</sub><sup>23</sup> -32.0 (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.30–7.23 (m, 5 H), 7.20–7.09 (m, 9 H), 7.08–7.04 (m, 1 H), 6.98–6.96 (m, 2 H),



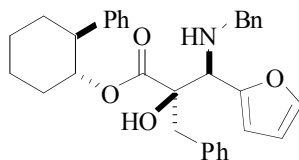
6.82–6.80 (m, 2 H), 4.81–4.76 (m, 1 H), 3.80 (s, 3 H), 3.45 (s, 1 H), 3.26 (s, 1 H), 3.04 (d,  $J = 13.5$  Hz, 1 H), 2.85–2.79 (m, 2 H), 2.60 (d,  $J = 13.5$  Hz, 1 H), 2.55 (d,  $J = 14.0$  Hz, 1 H), 2.06–2.03 (m, 1 H), 1.94–1.92 (m, 1 H), 1.79–1.73 (m, 2 H), 1.63–1.54 (m, 1 H), 1.44–1.35 (m, 2 H), 1.31–1.20 (m, 2 H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.7, 158.9, 143.5, 140.8, 136.2, 130.9, 130.4, 129.7, 128.6, 128.0, 127.9, 127.7, 127.5, 126.7, 126.5, 126.4, 113.5, 80.4, 80.0, 67.4, 55.2, 50.4, 49.8, 42.3, 34.0, 31.9, 25.9, 24.6; IR (film) 3494, 2934, 1725  $\text{cm}^{-1}$ . MS (ESI) 572.2778 (572.2777 calcd for  $\text{C}_{36}\text{H}_{39}\text{NO}_4$ ,  $\text{M} + \text{Na}^+$ ).

The enantiopurity of the title compound was assessed by conversion to the corresponding methyl ester (**S3**) through reaction with NaOMe using General Procedure B. This procedure afforded 20 mg (55%) of **S3**. The enantiopurity of the methyl ester was determined to be 90% ee by chiral HPLC analysis (chiracel AD 0.46 cm x 15 cm, 5% isopropanol/ hexanes, 0.2 mL/min, RT= 86.5 and 95.7 min).



**(+)-(2R,3S)-Methyl-2-benzyl-3-benzylamino-2-hydroxy-3-(4-methoxyphenyl)propanoate**

**(S3)**.  $[\alpha]_D^{23} +28.0$  (c 0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34–7.31 (m, 2 H), 7.28–7.27 (m, 2 H), 7.23–7.20 (m, 2 H), 7.19–7.15 (m, 4 H), 7.01–6.99 (m, 2 H), 6.95–6.93 (m, 2 H), 3.88 (s, 1 H), 3.84 (s, 3 H), 3.71 (s, 1 H), 3.68 (s, 3 H), 3.64 (s, 1 H), 3.45–3.34 (m, 2 H), 2.75 (d,  $J = 13.2$  Hz, 1 H), 2.39 (d,  $J = 13.2$  Hz, 1 H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  175.7, 159.2, 140.3, 135.8, 130.5, 130.3, 129.8, 128.4, 128.0, 126.8, 126.7, 113.7, 81.8, 65.3, 55.2, 52.4, 50.0, 43.2 (one carbon signal is absent due to incidental equivalence); IR (film) 3498, 3035, 1730  $\text{cm}^{-1}$ . MS (ESI) 428.1845 (428.1838 calcd for  $\text{C}_{25}\text{H}_{27}\text{NO}_4$ ,  $\text{M} + \text{Na}^+$ ).

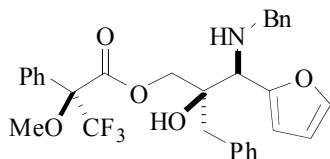


**(-)-(1R,2S,2'R,3'S)-2-Phenylcyclohexyl-2'-benzyl-3'-benzylamino-2'-furan-2-yl-2'-**

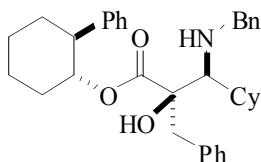
**hydroxypropanoate (8)**. The reaction of (-)-(1R,2S)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (52 mg, 0.16 mmol) with *N*-(furylidene)benzylamine,<sup>1</sup> (45 mg, 0.24 mmol) was conducted

according to General Procedure A using triethylamine as base to afford 56 mg (68%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through  $^1\text{H}$  NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 20:1 dr following purification.  $[\alpha]_{\text{D}}^{23} -9.5$  ( $c$  0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35–7.32 (m, 1 H), 7.31–7.23 (m, 3 H), 7.22–7.18 (m, 7 H), 7.17–7.09 (m, 4 H), 7.05–7.02 (m, 1 H), 6.30–6.29 (m, 1 H), 6.09–6.07 (m, 1 H), 4.85–4.80 (m, 1 H), 3.49 (s, 1 H), 3.45 (s, 1 H), 3.12 (d,  $J$  = 13.0 Hz, 1 H), 2.93 (d,  $J$  = 14.0 Hz, 1 H), 2.83–2.78 (m, 1 H), 2.69–2.65 (m, 2 H), 2.07–2.04 (m, 1 H), 1.94–1.90 (m, 1 H), 1.81–1.76 (m, 2 H), 1.60–1.47 (m, 2 H), 1.47–1.33 (m, 2 H), 1.32–1.24 (m, 1 H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.0, 153.0, 143.3, 141.9, 140.5, 135.8, 130.4, 128.6, 128.0, 127.9, 127.8, 127.4, 126.6, 126.5, 110.0, 108.7, 79.9, 79.8, 61.9, 50.6, 49.7, 41.8, 34.3, 31.9, 25.8, 24.6 (two carbon signals are absent due to incidental equivalence); IR (film) 3420, 2935, 1728  $\text{cm}^{-1}$ . MS (ESI) 510.2639 (510.2644 calcd for  $\text{C}_{33}\text{H}_{35}\text{NO}_4$ ,  $\text{M} + \text{Na}^+$ ).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S4**) using General Procedure C. This procedure afforded 6 mg (43%) of **S4**. The enantiopurity was determined to be 90% ee by  $^{19}\text{F}$  NMR analysis.



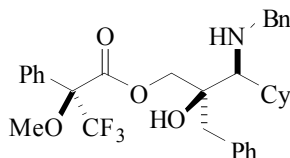
**(-)-(1S,2'R,3'S)-2'-Benzyl-3'-benzylamino-3'-furan-2-yl-2-hydroxy-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S4**).**  $[\alpha]_{\text{D}}^{23} -11.6$  ( $c$  0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.56–7.53 (m, 2 H), 7.44–7.43 (m, 1 H), 7.42–7.33 (m, 3 H), 7.29–7.27 (m, 2 H), 7.25–7.18 (m, 6 H), 7.14–7.12 (m, 2 H), 6.37–6.36 (m, 1 H), 6.04–6.03 (m, 1 H), 4.37–4.35 (m, 1 H), 3.90–3.85 (m, 3 H), 3.70–3.69 (m, 1 H), 3.45 (s, 3 H), 3.41–3.39 (m, 2 H), 2.75–2.72 (m, 1 H), 2.61–2.58 (m, 1 H);  $^{19}\text{F}$  NMR (376 MHz,  $(\text{CD}_3)_2\text{CO}$ )  $\delta$  -71.5; IR (film) 3376, 2936, 1751  $\text{cm}^{-1}$ . MS (ESI) 576.1972 (576.1974 calcd for  $\text{C}_{31}\text{H}_{30}\text{F}_3\text{NO}_5$ ,  $\text{M} + \text{Na}^+$ ).



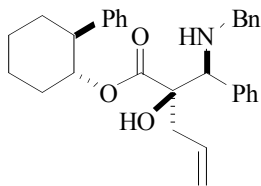
**(-)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-benzyl-3'-benzylamino-3'-cyclohexyl-2'-**

**hydroxypropanoate (9).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (43 mg, 0.13 mmol) with *N*-(cyclohexylmethylidene)benzylamine<sup>5</sup> (54 mg, 0.27 mmol, 2 equiv) was conducted according to General Procedure A using triethylamine as base to afford 46 mg (66%) of the title compound as a yellow oil. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_D^{23}$  -4.6 (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.37–7.35 (m, 2 H), 7.30–7.27 (m, 2 H), 7.25–7.19 (m, 6 H), 7.18–7.14 (m, 5 H), 4.58–4.53 (m, 1 H), 4.08 (s, 1 H), 3.03 (d, *J* = 13.5 Hz, 1 H), 2.95 (d, *J* = 14.0 Hz, 1 H), 2.86 (d, *J* = 12.5 Hz, 1 H), 2.71–2.64 (m, 2 H), 2.20 (s, 1 H), 1.89–1.86 (m, 1 H), 1.78–1.64 (m, 6 H), 1.62–1.60 (m, 3 H), 1.42–1.40 (m, 1 H), 1.35–1.25 (m, 3 H), 1.22–1.10 (m, 3 H), 1.06–0.88 (m, 3 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 175.2, 143.5, 141.1, 136.9, 130.3, 128.5, 128.2, 128.1, 127.9, 126.8, 126.7, 126.4, 79.6, 79.4, 67.5, 53.9, 49.7, 41.4, 38.8, 33.3, 32.5, 31.6, 27.0, 26.7, 26.3, 25.7, 24.5; IR (film) 3382, 2924, 1724 cm<sup>-1</sup>. MS (ESI) 526.3333 (526.3321 calcd for C<sub>35</sub>H<sub>43</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S5**) using General Procedure C. This procedure afforded 15 mg (71%) of **S5**. The enantiopurity was determined to be 93% ee by <sup>19</sup>F NMR analysis.

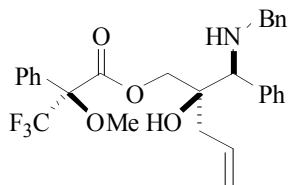


**(+)-(1*S*,2'*R*,3'*S*)-2'-Benzyl-3'-benzylamino-3'-cyclohexyl-2'-hydroxypropyl-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (S5).**  $[\alpha]_D^{23}$  +42.8 (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.56–7.54 (m, 2 H), 7.43–7.39 (m, 4 H), 7.34–7.29 (m, 4 H), 7.24–7.20 (m, 3 H), 7.15–7.13 (m, 2 H), 4.11–4.09 (m, 1 H), 3.88–3.80 (m, 2 H), 3.63–3.59 (m, 2 H), 3.53 (s, 3 H), 2.84–2.80 (m, 1 H), 2.64–2.61 (m, 2 H), 1.75–1.67 (m, 6 H), 1.30–1.22 (m, 6 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO) δ -71.0; IR (film) 3358, 2932, 1754 cm<sup>-1</sup>. MS (ESI) 570.2850 (570.2831 calcd for C<sub>33</sub>H<sub>38</sub>F<sub>3</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).



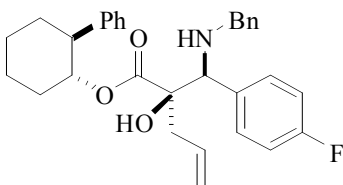
**(-)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-3'-benzylamino-2'-hydroxy-3'-phenylpent-4-enoate (10).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(allyloxy)acetate (**3b**) (46 mg, 0.17 mmol) with *N*-(benzylidene)benzylamine<sup>1</sup> (49 mg, 0.25 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 78 mg (70%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_D^{23} -36.7$  (c 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.28–7.21 (m, 7 H), 7.21–7.14 (m, 3 H), 7.10–7.08 (m, 2 H), 7.07–7.04 (m, 3 H), 5.44–5.37 (m, 1 H), 4.96–4.90 (m, 3 H), 3.38–3.36 (m, 2 H), 3.10 (d, *J* = 13.5 Hz, 1 H), 2.83–2.77 (m, 1 H), 2.71 (d, *J* = 13.5 Hz, 1 H), 2.40–2.32 (m, 2 H), 1.96–1.93 (m, 1 H), 1.89–1.79 (m, 3 H), 1.67–1.49 (m, 3 H), 1.42–1.33 (m, 2 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 174.5, 143.4, 140.6, 138.8, 131.9, 128.6, 128.1, 128.0, 127.9, 127.5, 127.4, 126.7, 126.6, 118.8, 79.8, 79.3, 67.6, 50.5, 49.7, 41.4, 34.1, 32.2, 25.8, 24.6 (one carbon signal is absent due to incidental equivalence); IR (film) 3494, 2935, 1727 cm<sup>-1</sup>. MS (ESI) 492.2903 (492.2515 calcd for C<sub>31</sub>H<sub>35</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S6**) using General Procedure C. This procedure afforded 16 mg (41%) of **S6**. The enantiopurity was determined to be 94% ee by <sup>19</sup>F NMR analysis.



**(+)-(2*S*,3'*R*,4'*S*)-3'-Allyl-4'-benzylamino-4'-phenyl-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S6**).**  $[\alpha]_D^{23} +11.0$  (c 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.60–7.56 (m, 2 H), 7.44–7.40 (m, 3 H), 7.37–7.29 (m, 5 H), 7.27–7.23 (m, 4 H), 7.15–7.13 (m, 1 H), 5.86–5.75 (m, 1 H), 5.10–5.08 (m, 1 H), 5.01–4.95 (m, 1 H), 4.55 (d, *J* = 11.2 Hz, 1 H), 3.90 (d, *J* = 11.6 Hz, 1 H), 3.75 (s, 1 H), 3.64 (d, *J* = 13.2 Hz, 1 H), 3.51 (s, 3 H), 3.38 (d, *J* = 13.2 Hz, 1 H),

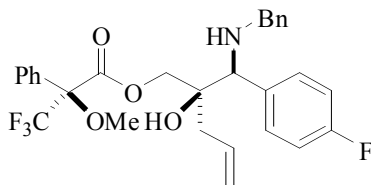
3.23 (s, br, 1 H), 2.20–2.15 (m, 2 H), 1.97–1.92 (m, 1 H);  $^{19}\text{F}$  NMR (376 MHz,  $(\text{CD}_3)_2\text{CO}$ )  $\delta$  –71.4; IR (film) 3397, 2949, 1752  $\text{cm}^{-1}$ . MS (ESI) 514.2204 (514.2205 calcd for  $\text{C}_{29}\text{H}_{30}\text{F}_3\text{NO}_4$ ,  $\text{M} + \text{Na}^+$ ).



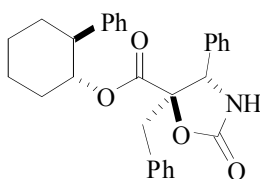
**(–)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-3'-benzylamino-3'-(*p*-fluorophenyl)-2'-**

**hydroxypent-4-enoate (11).** The reaction of (–)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(allyloxy)acetate (**3b**) (49 mg, 0.18 mmol) with *N*-(4-fluorophenylmethylidene)benzylamine<sup>1</sup> (57 mg, 0.27 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 59 mg (68%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through  $^1\text{H}$  NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_{\text{D}}^{23}$  –25.8 (*c* 0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.27–7.20 (m, 5 H), 7.19–7.14 (m, 2 H), 7.09–7.05 (m, 3 H), 6.98–6.91 (m, 4 H), 5.44–5.36 (m, 1 H), 4.98–4.91 (m, 3 H), 3.36–3.32 (m, 2 H), 3.09 (d,  $J$  = 13.0 Hz, 1 H), 2.82–2.76 (m, 1 H), 2.72 (d,  $J$  = 13.0 Hz, 1 H), 2.38–2.32 (m, 2 H), 1.96–1.93 (m, 1 H), 1.89–1.79 (m, 3 H), 1.65–1.59 (m, 2 H), 1.57–1.48 (m, 1 H), 1.42–1.33 (m, 2 H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.4, 163.4, 161.0, 143.4, 140.4, 134.5, 134.4, 131.7, 130.1, 128.6, 128.0, 127.4, 126.7, 118.9, 115.0 (d,  $J$  = 26.6 Hz), 79.7, 79.5, 67.9, 50.4, 49.7, 41.4, 34.1, 32.2, 25.8, 24.6; IR (film) 3366, 2931, 1730  $\text{cm}^{-1}$ . MS (ESI) 488.2595 (488.2601 calcd for  $\text{C}_{31}\text{H}_{34}\text{FNO}_3$ ,  $\text{M} + \text{Na}^+$ ).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S7**) using General Procedure C. This procedure afforded 27 mg (50%) of **S7**. The enantiopurity was determined to be 94% ee by  $^{19}\text{F}$  NMR analysis.

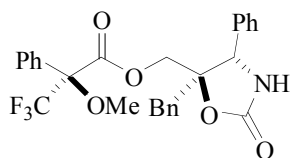


**(+)-(2*S*,3'*R*,4'*S*)-3'-Allyl-4'-benzylamino-4'-(*p*-fluorophenyl)-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (S7).**  $[\alpha]_D^{23} +5.0$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56–7.49 (m, 2 H), 7.45–7.36 (m, 3 H), 7.31–7.24 (m, 3 H), 7.21–7.16 (m, 3 H), 7.11–7.07 (m, 1 H), 7.02–6.95 (m, 2 H), 5.82–5.71 (m, 1 H), 5.10–5.09 (m, 1 H), 5.00–4.96 (m, 1 H), 4.54 (t, *J* = 11.6 Hz, 1 H), 4.04 (d, *J* = 11.2 Hz, 0.5 H), 3.90 (d, *J* = 14.5 Hz, 0.5 H), 3.72 (s, 1 H), 3.61–3.56 (m, 2 H), 3.50 (s, 3 H), 3.39–3.32 (m, 1 H), 2.19–2.11 (m, 1 H), 1.98–1.85 (m, 2 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO)  $\delta$  –71.3; IR (film) 3386, 2929, 1750 cm<sup>–1</sup>. MS (ESI) 532.2110 (532.2111 calcd for C<sub>29</sub>H<sub>29</sub>F<sub>4</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

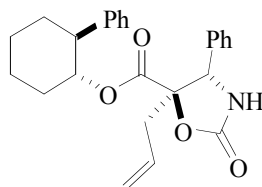


**(–)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-benzyl-2'-oxo-3'-phenyloxazolidine-5'-carboxylate (13).** The reaction of (–)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (56 mg, 0.18 mmol) with *N*-(*tert*-butoxycarbonyl)benzylamine<sup>2</sup> (55 mg, 0.27 mmol) was conducted according to General Procedure A using triethylamine as base to afford 52 mg (97%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_D^{23} -64.0$  (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.37–7.29 (m, 7 H), 7.23–7.14 (m, 4 H), 7.12–7.08 (m, 2 H), 7.00–6.98 (m, 2 H), 5.16–5.11 (m, 1 H), 5.03 (s, 1 H), 3.69 (s, 1 H), 2.85–2.80 (m, 1 H), 2.45 (d, *J* = 15.0 Hz, 1 H), 2.33 (d, *J* = 15.0 Hz, 1 H), 2.00–1.98 (m, 1 H), 1.84–1.78 (m, 3 H), 1.56–1.48 (m, 1 H), 1.45–1.32 (m, 3 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  169.1, 158.1, 143.1, 135.8, 134.1, 129.9, 129.1, 128.9, 128.8, 128.0, 127.4, 127.2, 127.1, 86.5, 62.1, 50.4, 39.4, 34.8, 31.9, 25.7, 24.7 (two carbon signals are absent due to incidental equivalence); IR (film) 3270, 2935, 1766 cm<sup>–1</sup>. MS (ESI) 478.2001 (478.1994 calcd for C<sub>29</sub>H<sub>29</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S8**) using General Procedure C. This procedure afforded 16 mg (64%) of **S8**. The enantiopurity was determined to be 94% ee by <sup>19</sup>F NMR analysis.

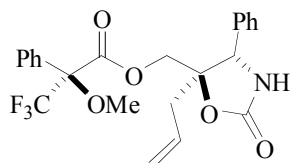


**(+)-(2*S*,4'*R*,5'*S*)-4'-Benzyl-4'-oxo-5'-phenyloxazolidine-5'-carboxylate-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (S8).**  $[\alpha]^{23}_{\text{D}} +8.5$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.57–7.56 (m, 2 H), 7.48–7.30 (m, 7 H), 7.25–7.15 (m, 4 H), 7.03–7.00 (m, 2 H), 5.31 (s, 1 H), 4.79 (s, 1 H), 4.28 (d, *J* = 11.6 Hz, 1 H), 4.17 (d, *J* = 12.0 Hz, 1 H), 3.56 (s, 3 H), 2.59 (d, *J* = 14.8 Hz, 1 H), 2.12 (d, *J* = 14.4 Hz, 1 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO)  $\delta$  –71.1; IR (film) 3387, 2921, 1760 cm<sup>–1</sup>. MS (ESI) 522.1497 (522.1504 calcd for C<sub>27</sub>H<sub>24</sub>F<sub>3</sub>NO<sub>5</sub>, M + Na<sup>+</sup>).

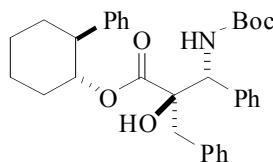


**(–)-(1*R*,2*S*,2'*R*,3'*S*)-2-Phenylcyclohexyl-2'-allyl-2'-oxo-3'-phenyloxazolidine (14).** The reaction of (–)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(allyloxy)acetate (**3b**) (47 mg, 0.17 mmol) with *N*-(*tert*-butoxycarbonyl)benzylamine<sup>2</sup> (53 mg, 0.26 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 45 mg (62%) of the title compound as a colorless oil. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]^{23}_{\text{D}} -6.1$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.35–7.30 (m, 7 H), 7.22–7.17 (m, 1 H), 6.99–6.97 (m, 2 H), 5.42–5.32 (m, 1 H), 5.26–5.20 (m, 1 H), 5.09 (s, 1 H), 4.91–4.83 (m, 2 H), 3.95 (s, 1 H), 2.86–2.80 (m, 1 H), 2.19–2.16 (m, 1 H), 2.06–2.00 (m, 1 H), 1.99–1.89 (m, 2 H), 1.85–1.81 (m, 1 H), 1.75–1.63 (m, 1 H), 1.60–1.34 (m, 4 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  169.6, 143.0, 135.7, 130.0, 129.1, 128.8, 127.5, 127.1, 126.6, 119.8, 86.1, 77.8, 61.7, 50.1, 38.1, 34.6, 32.1, 25.7, 24.7 (two carbon signals are absent due to incidental equivalence); IR (film) 3295, 2934, 1766 cm<sup>–1</sup>. MS (ESI) 428.1829 (428.1838 calcd for C<sub>25</sub>H<sub>27</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S9**) using General Procedure C. This procedure afforded 71 mg (53%) of **S9**. The enantiopurity was determined to be 93% ee by  $^{19}\text{F}$  NMR analysis.



**(+)-(2*S*,4'*R*,5'*S*)-4'-Allyl-4'-oxo-5'-phenyloxazolidine-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S9**).**  $[\alpha]_D^{23} +9.0$  (*c* 0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58–7.56 (m, 2 H), 7.47–7.44 (m, 3 H), 7.41–7.35 (m, 3 H), 7.16–7.14 (m, 2 H), 5.62–5.55 (m, 1 H), 5.35 (s, 1 H), 5.07 (d, *J* = 10.5 Hz, 1 H), 4.91–4.88 (m, 1 H), 4.74 (s, 1 H), 4.49 (d, *J* = 12.0 Hz, 1 H), 4.41 (d, *J* = 11.5 Hz, 1 H), 3.58 (s, 3 H), 2.10–2.06 (m, 1 H), 1.72–1.67 (m, 1 H);  $^{19}\text{F}$  NMR (376 MHz,  $(\text{CD}_3)_2\text{CO}$ )  $\delta$  –71.3; IR (film) 3273, 2926, 1760  $\text{cm}^{-1}$ . MS (ESI) 472.1336 (472.1348 calcd for  $\text{C}_{23}\text{H}_{22}\text{F}_3\text{NO}_5$ ,  $\text{M} + \text{Na}^+$ ).

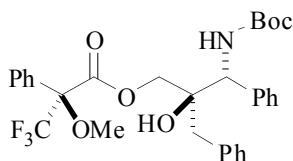


**(–)-(1*R*,2*S*,2'*R*,3'*R*)-2-Phenylcyclohexyl-2'-benzyl-3'-[(*tert*-butoxycarbonyl)amino]-2'-hydroxy-3'-phenylpropanoate (**16**).** The reaction of (–)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (53 mg, 0.16 mmol) with *N*-*tert*-butoxycarbonyl- $\alpha$ -(phenylsulfonyl)benzylamine<sup>2</sup> (85 mg, 0.25 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 50 mg (58%) of the title compound as a white solid, m.p. 160–162 °C. The diastereoselectivity of the transformation could not be determined through  $^1\text{H}$  NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with >20:1 dr following purification.  $[\alpha]_D^{23} -8.0$  (*c* 0.20,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.46–7.38 (m, 1 H), 7.31–7.27 (m, 3 H), 7.22–7.21 (m, 3 H), 7.15–7.13 (m, 5 H), 7.08–7.06 (m, 2 H), 6.43–6.41 (m, 1 H), 5.49–5.46 (m, 1 H), 4.89–4.84 (m, 1 H), 4.65–4.63 (m, 1 H), 3.12–3.08 (m, 1 H), 3.03–3.00 (m, 1 H), 2.98 (s, 1 H), 2.92–2.86 (m, 1 H), 2.03–2.00 (m, 1 H), 1.91–1.88 (m, 1 H), 1.81–1.63 (m, 1 H), 1.58 (s, 1 H), 1.53 (s, 9 H), 1.45–1.35 (m, 2 H), 1.30–1.21 (m, 2 H);  $^{13}\text{C}$

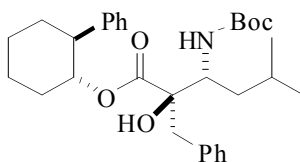


NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  173.3, 154.7, 143.1, 137.5, 135.2, 130.6, 129.2, 127.9, 127.8, 127.6, 127.5, 127.4, 126.9, 80.9, 79.5, 60.0, 49.2, 33.8, 31.8, 28.5, 24.5 (four carbon signals are absent due to incidental equivalence); IR (film) 3429, 2934, 1718 cm<sup>-1</sup>. MS (ESI) 552.2718 (552.2726 calcd for C<sub>33</sub>H<sub>39</sub>NO<sub>5</sub>, M + Na<sup>+</sup>).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S10**) using General Procedure C. This procedure afforded 21.3 mg (51%) of **S10**. The enantiopurity was determined to be 96% ee by <sup>19</sup>F NMR analysis.



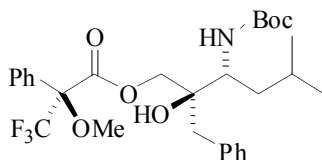
(-)-(1*S*,2'*R*,3'*R*)-2'-Benzyl-3'-[(*tert*-butoxycarbonyl)amino]-2'-hydroxy-3'-phenylpropyl-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S10**). [ $\alpha$ ]<sub>D</sub><sup>23</sup> -26.0 (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.67–7.66 (m, 2 H), 7.52–7.46 (m, 3 H), 7.36–7.29 (m, 5 H), 7.21–7.16 (m, 3 H), 6.84–6.81 (m, 2 H), 5.58–5.55 (m, 1 H), 4.82–4.80 (m, 1 H), 4.23 (d, *J* = 11.2 Hz, 1 H), 3.70 (s, 3 H), 3.67–3.60 (m, 1 H), 2.72–2.69 (m, 1 H), 2.39–2.36 (m, 1 H), 1.54 (s, 1 H), 1.40 (s, 9 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO)  $\delta$  -70.9; IR (film) 3432, 2932, 1752 cm<sup>-1</sup>. MS (ESI) 596.2254 (596.2236 calcd for C<sub>31</sub>H<sub>34</sub>F<sub>3</sub>NO<sub>6</sub>, M + Na<sup>+</sup>).



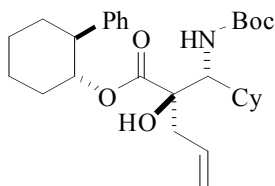
(-)-(1*R*,2*S*,2'*R*,3'*R*)-2-Phenylcyclohexyl-2'-benzyl-3'-[(*tert*-butoxycarbonyl)amino]-2'-hydroxy-5'-methylhexanoate (**17**). The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3a**) (49 mg, 0.15 mmol) with *N*-*tert*-butoxycarbonyl-3-methyl-1-(phenylsulfonyl)butylcarbamate<sup>3</sup> (75 mg, 0.23 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 62 mg (81%) of the title compound as a white foam. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H

NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 20:1 dr following purification.  $[\alpha]_D^{23}$   $-6.8$  ( $c$  0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38–7.35 (m, 3 H), 7.30–7.26 (m, 2 H), 7.25–7.19 (m, 5 H), 4.94–4.88 (m, 1 H), 3.92–3.90 (m, 1 H), 3.58–3.54 (m, 1 H), 3.27–3.20 (m, 1 H), 3.12 (d,  $J$  = 13.5 Hz, 1 H), 2.92 (d,  $J$  = 14.0 Hz, 1 H), 2.88–2.83 (m, 1 H), 2.06–2.03 (m, 1 H), 1.99–1.97 (m, 1 H), 1.86–1.82 (m, 1 H), 1.81–1.77 (m, 1 H), 1.57–1.54 (m, 2 H), 1.53 (s, 9 H), 1.41–1.37 (m, 5 H), 0.74–0.72 (m, 3 H), 0.67–0.66 (m, 3 H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.1, 155.8, 143.4, 135.8, 130.6, 129.4, 127.8, 127.3, 126.7, 80.2, 79.6, 78.9, 54.7, 49.7, 41.6, 37.8, 34.6, 32.2, 28.5, 28.4, 25.6, 24.7, 23.3, 20.9 (one carbon signal is absent due to incidental equivalence); IR (film) 3420, 2923, 1718  $\text{cm}^{-1}$ . MS (ESI) 532.3027 (532.3039 calcd for  $\text{C}_{31}\text{H}_{43}\text{NO}_5$ ,  $\text{M} + \text{Na}^+$ ).

The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S11**) using General Procedure C. This procedure afforded 12.2 mg (53%) of **S11**. The enantiopurity was determined to be 91% ee by  $^{19}\text{F}$  NMR analysis.

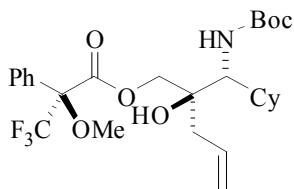


**(–)-(1*S*,2'*R*,3'*R*)-2'-Benzyl-3'-[(*tert*-butoxycarbonyl)amino]-2'-hydroxy-5'-methylhexyl-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S11**).**  $[\alpha]_D^{23}$   $-28.0$  ( $c$  0.10,  $\text{CH}_2\text{Cl}_2$ ).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58–7.53 (m, 2 H), 7.41–7.40 (m, 3 H), 7.20–7.19 (m, 3 H), 7.04–7.03 (m, 2 H), 4.48–4.42 (m, 1 H), 4.09–4.05 (m, 1 H), 3.90–3.87 (m, 1 H), 3.71–3.63 (m, 1 H), 3.57 (s, 3 H), 3.52 (s, 1 H), 2.91 (s, 1 H), 2.80–2.76 (m, 1 H), 2.69–2.65 (m, 1 H), 1.50 (s, 2 H), 1.39 (s, 9 H), 0.88 (d,  $J$  = 6.8 Hz, 3 H), 0.78 (d,  $J$  = 6.4 Hz, 3 H);  $^{19}\text{F}$  NMR (376 MHz,  $(\text{CD}_3)_2\text{CO}$ )  $\delta$   $-70.9$ ; IR (film) 3420, 2958, 1751  $\text{cm}^{-1}$ . MS (ESI) 576.2545 (576.2549 calcd for  $\text{C}_{29}\text{H}_{38}\text{F}_3\text{NO}_6$ ,  $\text{M} + \text{Na}^+$ ).

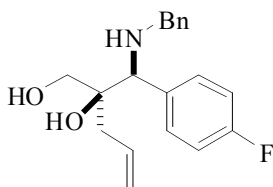


**(-)-(1*R*,2*S*,2'*R*,3'*R*)-2-Phenylcyclohexyl-3'-[(*tert*-butoxycarbonyl)amino]-3'-cyclohexyl-2'-hydroxypent-4-enoate (18).** The reaction of (-)-(1*R*,2*S*)-2-phenylcyclohexyl-2'-(benzyloxy)acetate (**3b**) (44 mg, 0.16 mmol) with *N-tert*-butoxycarbonyl- $\alpha$ -cyclohexyl(phenylsulfonyl)methylcarbamate<sup>3</sup> (85 mg, 0.24 mmol) was conducted according to General Procedure A using diisopropylethylamine as base to afford 49 mg (63%) of the title compound as a colorless oil. The diastereoselectivity of the transformation could not be determined through <sup>1</sup>H NMR analysis of the crude reaction mixture prior to purification due to signal overlap with boron-containing byproducts; the isolated product was obtained with 20:1 dr following purification. [ $\alpha$ ]<sub>D</sub><sup>23</sup> -21.4 (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.38–7.31 (m, 2 H), 7.29–7.24 (m, 2 H), 7.20–7.16 (m, 1 H), 5.52–5.46 (m, 1 H), 5.08–5.02 (m, 3 H), 4.89–4.83 (m, 1 H), 4.61 (d, *J* = 11.0 Hz, 1 H), 3.22 (s, 1 H), 2.80–2.75 (m, 1 H), 2.52–2.48 (m, 1 H), 2.35–2.25 (m, 3 H), 1.98–1.95 (m, 1 H), 1.89–1.86 (m, 1 H), 1.82–1.79 (m, 2 H), 1.72–1.70 (m, 1 H), 1.66–1.60 (m, 2 H), 1.54 (s, 9 H), 1.44–1.38 (m, 7 H), 1.38–1.26 (m, 3 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  175.3, 155.6, 143.3, 131.4, 129.0, 127.6, 127.2, 119.8, 80.5, 78.9, 60.2, 49.5, 42.3, 36.9, 33.9, 32.0, 31.3, 28.5, 26.2, 25.9, 25.7, 25.3, 24.5; IR (film) 3420, 2923, 1718 cm<sup>-1</sup>. MS (ESI) 508.3029 (508.3039 calcd for C<sub>29</sub>H<sub>43</sub>NO<sub>5</sub>, M + Na<sup>+</sup>).

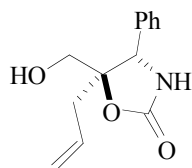
The enantiopurity of the title compound was assessed by conversion to the corresponding Mosher ester (**S12**) using General Procedure C. This procedure afforded 13.4 mg (67%) of **S12**. The enantiopurity was determined to be 90% ee by <sup>19</sup>F NMR analysis.



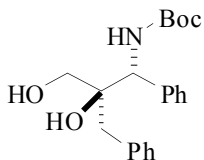
**(-)-(1*S*,2'*R*,3'*R*)-3'-[(*tert*-Butoxycarbonyl)amino]-3'-cyclohexyl-2'-hydroxypent-4'-enyl-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (S12).** [ $\alpha$ ]<sub>D</sub><sup>23</sup> -48.0 (*c* 0.20, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56–7.52 (m, 2 H), 7.44–7.40 (m, 3 H), 5.75–5.65 (m, 1 H), 5.19–5.12 (m, 1 H), 4.91–4.88 (m, 1 H), 4.23–4.11 (m, 1 H), 3.56 (s, 3 H), 3.53–3.50 (m, 1 H), 2.31–2.29 (m, 2 H), 2.04 (s, 1 H), 1.88–1.81 (m, 1 H), 1.74–1.71 (m, 3 H), 1.68–1.61 (m, 1 H), 1.40 (s, 9 H), 1.23–1.14 (m, 4 H), 1.13–1.04 (m, 2 H), 1.00–0.88 (m, 1 H), 0.86–0.84 (m, 1 H); <sup>19</sup>F NMR (376 MHz, (CD<sub>3</sub>)<sub>2</sub>CO)  $\delta$  -71.4; IR (film) 3398, 2923, 1719 cm<sup>-1</sup>. MS (ESI) 552.2542 (552.2549 calcd for C<sub>27</sub>H<sub>38</sub>F<sub>3</sub>NO<sub>6</sub>, M + Na<sup>+</sup>).



**(+)-(1*S*,2*R*)-3-Benzylamino-3-(*p*-fluorophenyl)-2-hydroxypent-4-ene-1-ol (20).** The reaction of (–)-(1*R*,2*S*,2′*R*,3′*S*)-2-Phenylcyclohexyl-3′-benzylamino-3′-(*p*-fluorophenyl)-2′-hydroxypent-4-enoate (**11**) (49 mg, 0.10 mmol) with lithium aluminum hydride (0.20 mL, 0.20 mmol, 1 M in THF) was conducted according to General Procedure C to afford 31 mg (97%) of the title compound as an oil.  $[\alpha]_D^{23} +14.8$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.37–7.32 (m, 4 H), 7.28–7.26 (m, 2 H), 7.23–7.13 (m, 1 H), 7.09–6.96 (m, 2 H), 5.70–5.65 (m, 1 H), 5.02–5.00 (m, 1 H), 4.92–4.89 (m, 1 H), 3.66–3.61 (m, 3 H), 3.56–3.55 (m, 1 H), 3.46–3.42 (m, 1 H), 2.08–1.94 (m, 2 H), 1.84–1.77 (m, 2 H), 1.60 (s, br, 1 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 132.5, 130.2, 128.7, 128.4, 127.9, 127.5, 118.9, 115.4 (d, *J*= 26.6 Hz), 74.3, 69.5, 67.7, 62.1, 50.9, 40.5, 25.7; IR (film) 3370, 2924 cm<sup>−1</sup>. MS (ESI) 316.1705 (316.1713 calcd for C<sub>19</sub>H<sub>22</sub>FNO<sub>2</sub>, M + Na<sup>+</sup>).



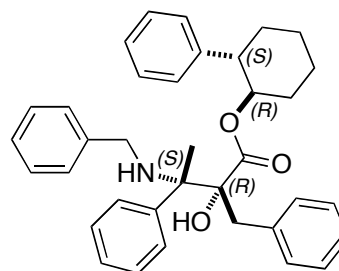
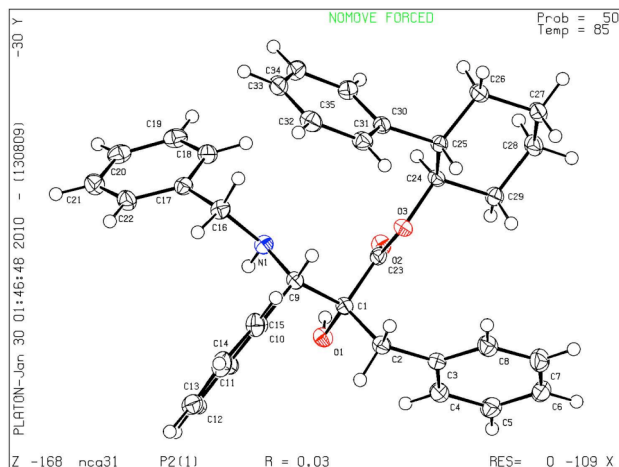
**(+)-(1*S*,2*R*)-2-Allyl-2-hydroxymethyl-1-phenyloxazolidin-2-one (21).** The reaction of (–)-(1*R*,2*S*,2′*R*,3′*S*)-2-Phenylcyclohexyl-2′-allyl-2′-oxo-3′-phenyloxazolidine (**14**) (34 mg, 0.08 mmol) with lithium aluminum hydride (0.17 mL, 0.17 mmol, 1 M in THF) was conducted according to General Procedure C to afford 13 mg (65%) of the title compound as an oil.  $[\alpha]_D^{23} +45.0$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.42–7.34 (m, 3 H), 7.33–7.27 (m, 2 H), 5.60–5.49 (m, 1 H), 5.44, (s, br, 1 H), 5.16 (s, 1 H), 5.01–4.99 (m, 1 H), 4.91–4.87 (m, 1 H), 3.91 (d, *J*= 12.4 Hz, 1 H), 3.63 (d, *J*= 12.4 Hz, 1 H), 2.32 (s, br, 1 H), 2.21–2.04 (m, 1 H), 1.76–1.67 (m, 1 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 176.0, 158.2, 136.3, 131.3, 128.9, 127.1, 119.5, 86.9, 64.9, 60.0, 37.7; IR (film) 3307, 2930, 1748 cm<sup>−1</sup>. MS (ESI) 233.1059 (233.1052 calcd for C<sub>13</sub>H<sub>15</sub>NO<sub>3</sub>, M + Na<sup>+</sup>).



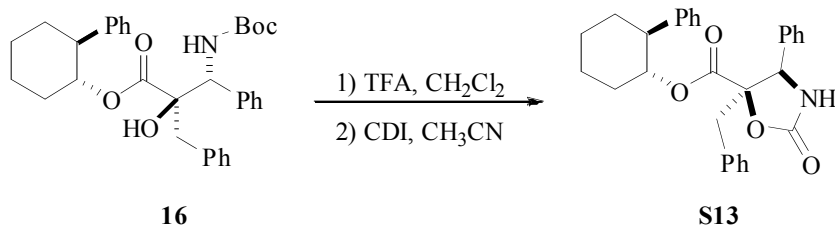
**(+)-(1R,2R)-[(*tert*-Butoxycarbonyl)amino]-2-benzyl-1-phenylpropane-2,3-diol (22).** The reaction of (–)-(1*R*,2*S*,2′*R*,3′*R*)-2-Phenylcyclohexyl-2′-benzyl-3′-[(*tert*-butoxycarbonyl)amino]-2′-hydroxy-3′-phenylpropanoate (**16**) (50 mg, 0.10 mmol) with lithium aluminum hydride (0.38 mL, 0.38 mmol, 1 M in THF) was conducted according to General Procedure C to afford 26 mg (77%) of the title compound as an oil.  $[\alpha]_D^{23} +16.4$  (*c* 0.10, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.48–7.45 (m, 2 H), 7.41–7.32 (m, 4 H), 7.29–7.27 (m, 1 H), 7.25–7.22 (m, 1 H), 7.14–7.13 (m, 2 H), 5.40–5.38 (m, 1 H), 4.85–4.83 (m, 1 H), 3.57–3.55 (m, 1 H), 3.40–3.36 (m, 1 H), 3.18–3.13 (m, 1 H), 2.97–2.94 (m, 1 H), 2.29–2.26 (m, 1 H), 1.63 (s, 1 H), 1.45 (s, 9 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 157.0, 138.4, 135.9, 130.6, 128.9, 128.6, 128.5, 127.9, 126.8, 80.5, 62.9, 58.4, 40.1, 28.3 (one carbon signal is absent due to incidental equivalence); IR (film) 3412, 2924, 1684 cm<sup>−1</sup>. MS (ESI) 380.1842 (380.1838 calcd for C<sub>21</sub>H<sub>27</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

## Assignment of Stereochemistry

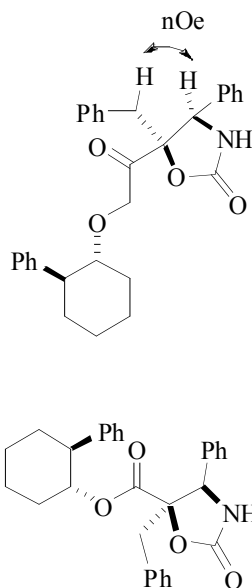
**Stereochemical Assignment of (4).** Stereochemical assignment of **4** was accomplished by single-crystal x-ray analysis of product that had been recrystallized from dichloromethane/hexanes. The ORTEP of **4** indicated this material had the **1*R*,2*S*,2′*R*,3′*S*** stereochemical configuration. The data for **4** (CCDC 763869) can be obtained free of charge from The Cambridge Crystallographic Data Centre via the internet at [www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk).



**Stereochemical Analysis of Amino Alcohol (16).** Amino alcohol **16** was converted to derivative (**S13**) via deprotection of the Boc group followed by conversion to the oxazolidin-2-one with CDI.



The relative stereochemistry of (**S13**) was assigned based on the nOe signals depicted below



**(-)-(1R,2S,2'S,3'S)-2-Phenylcyclohexyl-2'-benzyl-2'-oxo-3'-phenyloxazolidine-4'-**

**carboxylate (S13).** A flame-dried flask was charged with amino alcohol **16** (32 mg, 0.06 mmol, 1.0 equiv) in dichloromethane (0.6 mL) and cooled to 0 °C. Trifluoroacetic acid (0.6 mL) was added dropwise, and the resulting solution was warmed to rt and stirred until the starting material had been completely consumed judged by TLC analysis (ca 1 h). Aqueous sodium carbonate (1.5 mL) and dichloromethane (8 mL) were added, and the resulting mixture was transferred to a separatory funnel. The layers were separated, and the organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product (24 mg, 0.06 mmol, 94%) was added as a solution in acetonitrile (0.6 mL) to a flame dried two-neck flask fitted with a reflux condenser. Carbonyldiimidazole (10 mg, 0.06 mmol, 1.1 equiv) was added and the

reaction mixture was heated to reflux until the starting material was completely consumed as judged by TLC analysis (ca 14 h). The reaction mixture was cooled to rt and concentrated *in vacuo*. The crude residue was diluted with H<sub>2</sub>O (1 mL/mmol substrate), and extracted with dichloromethane (3 x 3 mL/mmol substrate). The phases were separated and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography on silica gel affording 22 mg (84%) of the title compound as a white foam.  $[\alpha]_D^{23}$  -36.2 (*c* 0.1, CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.36–7.28 (m, 5 H), 7.24–7.21 (m, 5 H), 7.20–7.09 (m, 5 H), 4.97–4.93 (m, 1 H), 3.54 (s, 1 H), 3.40 (s, 1 H), 2.85–2.78 (m, 1 H), 2.68 (d, *J* = 14.0 Hz, 1 H), 2.28 (d, *J* = 13.6 Hz, 1 H), 2.03–1.91 (m, 2 H), 1.84–1.78 (m, 3 H), 1.63–1.52 (m, 2 H), 1.49–1.34 (m, 1 H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 174.4, 143.4, 141.4, 136.1, 130.1, 128.7, 128.3, 127.8, 127.6, 127.0, 126.6, 80.7, 79.0, 61.4, 50.2, 42.3, 34.1, 32.3, 25.7, 24.7, 24.4, 24.1, 21.0; IR (film) 3391, 2932, 1734 cm<sup>-1</sup>. MS (ESI) 478.2017 (478.1994 calcd for C<sub>29</sub>H<sub>29</sub>NO<sub>4</sub>, M + Na<sup>+</sup>).

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